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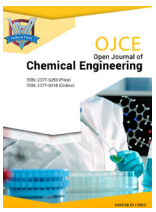

ARTICLE

CHEMICAL COMPOSITIONS AND FUNCTIONS OF CAMPHOR TREE VOLATILES

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ABSTRACT

Camphor tree is the dominant tree species of evergreen broad - leaved forest in subtropical regions of China, and has important ecological and economic effects. A large number of scholars have studied the camphor extract. In this paper, the camphor tree powder volatiles were analyzed by fourier transform infrared spectroscopy (FT-IR), thermogravimetry (TG), pyrolisis-gas chromatography-mass spectrometry (Py-GC-MS), thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS). Experiments show that camphor volatile composition contains alcohols, phenol, ester, aromatic, amine. Camphor tree powder was detected in PY-GC-MS analysis, 50 peaks were detected and 12 chemical constituents were identified and the peak area accounted for 20.78% of the total peak area. Camphor tree powder was detected in TDS-GC-MS analysis, 9 chemical constituents were identified in 34 peaks.

KEYWORDS

Camphor tree; volatiles; FT-IR; Py-GC-MS; TD-GC-MS; TG.

1. INTRODUCTION

Camphor tree is a family of camphor and genus of camphor evergreen trees. Produced in southern China, southwest provinces and Japan, North Korea and other countries. Other countries are often introduced cultivation. As the important timber and special economic tree species in China's subtropical evergreen broad-leaved forest, camphor not only has a majestic appearance, green throughout the year, shade, expelling insect and sterilization in terms of landscaping, but also used to make furniture for its excellent material. Some chemicals that can be applied to foodstuff, leechdom, chemical and other fields can be extracted from camphor's leaves, branches, roots, bark and fruit.

Because camphor trees occupy an important position in China's forestry, many scholars have done a lot of research, and has achieved very fruitful results, for example cultivar maintenance, rapid propagation by cutting tissue culture, artificial forest dynamic, pest control, active ingredient and so on [1]. There are many scholars in China to study and explore the chemical composition of camphor extract and its application, but the lack of the volatiles of camphor trees related research. This article starts from the volatiles of camphor trees, the chemical constituents of the volatile materials of camphor wood were analyzed by fourier transform infrared spectroscopy (FT-IR), thermogravimetry (TG), pyrolisis-gas chromatography-mass spectrometry (Py-GC-MS), thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS). And explore its functional application value.

2. MATERIAL AND METHODS

2.1 Experimental materials

The experimental material is taken from camphor wood in Central South University of Forestry and Technology. And crushed by micro plant grinding machine.

2.2 Experimental methods

2.2.1 FT-IR analysis

First put the camphor powder into drying oven, set the temperature to 100°C and dry for 6 hours to evaporate the free water from the sample. Then make the dried camphor powder through a 200-mesh screen. This experiment uses pure KBr as a solid dispersion medium. The finely ground camphor powder was dispersed in KBr at 1: 100, and be measured after pressed with a tabletting device into a transparent sheet. The range of the spectrum is set to 400 cm^{-1}-4000 cm^{-1} [2].

2.2.2 Py-GC-MS analysis

The material of the experiment was untreated camphor tree powder which is passed through a 200-mesh screen. Carrier gas is high purity He; pyrogenation temperature is 500°C, heating rate is 20°C/min, Pyrolysis time is 15s; chromatographic column is TR-5MS; Capillary column is (30 m×0.25 mm×0.25 μm); Set the transfer line of pyrolysis product and the injection valve temperature to 300°C; Shunt mode, split ratio of 1:60, shunt rate of 50 mL/min. Set the temperature of GC program start at 40°C and keep for 2 min, rises to 120°C at a rate of 5°C/min, then rises to 200°C at a rate of 10°C/min and keep for 15 min. The temperature of ion

source is 280℃, scanning range is 28 amu-500 amu [3].

2.2.3 TD-GC-MS analysis

The material of the experiment was untreated camphor tree powder which is passed through a 200-mesh screen. First put the camphor powder into drying oven, set the temperature to 100℃ and dry for 6 hours to evaporate the free water from the sample.

Set the temperature of TDS program start at 30℃ and keep for 1 min, rises to 100℃ at a rate of 10℃/min and keep for 5 min, then rises to 200℃ at a rate of 10℃/min, transmission line temperature is 230℃. Cold injection system starts at -50℃, rises to 230℃ at a rate of 10℃/min and keep for 1 min [4].

GC-MS: The temperature of GC program starts at 50℃ and keep for 2 min, rises to 250℃ at a rate of 8℃/min, then rises to 300℃ at a rate of 5℃/min and keep for 3 min. MS program scanning quality range is 30 amu-600 amu. ionization mode is EI, ionization voltage is 70 eV, ionization current is 150 μA. Ion source temperature is 230℃, quadrupole temperature is 150℃ [5-7].

2.2.4 TG analysis

First put the camphor powder into drying oven,set the temperature to 100℃ and dry for 6 hours to oven dry. Then make the dried camphor powder through a 200-mesh screen. The gas atmosphere is nitrogen, gas flow is 60 mL/min; temperature range is 30℃-250℃, heating rate is 5℃/min.

3. RESULTS AND DISCUSSION

3.1 Analysis of FT-IR

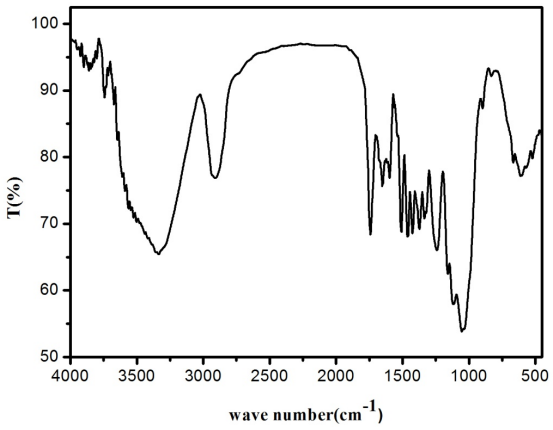

Figure 1: FT-IR spectra.

From Figure 1 and Table 1, 3335cm^{-1} is stretching vibration absorbance peak of alcohol molecular and phenol molecular (-OH). This wave number forms a broad and strong absorption peak because alcohols and phenols generally form intermolecular hydrogen bonds, weakening the O-H bond strength, thereby reducing the absorption frequency [8].

2911 cm^{-1} is stretching vibration absorbance peak of alkane molecular (C-H). 1742 cm^{-1} is stretching vibration absorbance peak of ketones molecular (C=O). 1597 cm^{-1}, 1508 cm^{-1},1464 cm^{-1} is framework ring stretching vibration absorbance peak of benzene ring. 1335cm^{-1} is stretching vibration absorbance peak of amine molecular (C-N). 1055 cm^{-1} is stretching vibration absorbance peak of alcohol, phenol and ester molecular (C-O). The main chemical composition of wood is the substances of constitute the wood cell wall and intercellular layer. Composed of three kinds of polymer compounds, including cellulose, hemicellulose and lignin which total amount of wood accounted for more than 90%. Except these main compositions, the volatile matter which we need to study only a small amount, therefore the absorbance peak of volatile matter will be affected to a large extent.

FT-IR absorbance peak of untreated camphor tree powder mainly concentrated in 3700 cm^{-1}-3000 cm^{-1}, 2900 cm^{-1}-2700 cm^{-1}, 1750 cm^{-1}-1000 cm^{-1}, corresponding chemical composition of absorb infrared radiation include alcohol, phenol, ester, aromatic, amine.

3.2 Analysis of Py-GC-MS

In general, infrared spectra are only suitable for the analysis of pure compounds. Although the above experiment, the chemical composition of volatile matter contained in camphor tree powder was analyzed, there are some limitations to identify only by FT-IR. It is difficult to identify those synthetic fibers containing the same chemical characteristic groups. Therefore, we continued to do Py-GC-MS analysis because Py-GC-MS can qualitative and quantitative analysis to small molecules of after high molecular weight pyrolysis [9].

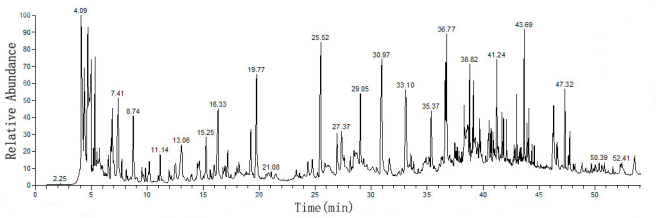

Figure 2: Total ion chromatograms by Py-GC-MS.

According to the results of Py-GC-MS analysis, 50 peaks were detected and 12 chemical constituents were identified and the peak area accounted for 20.78% of the total peak area. Sorting the detected component by peak area ratio from high to low are: Phenol, 2-methoxy- (3.93%), Propanoic acid, 2-oxo-, methyl ester (3.05%),2-Propanone, 1-hydroxy (2.62%), 1,2-Cyclopentanedione (2.34%), Phenol, 4-ethyl-2-methoxy- (2.29%), Phenol, 2,6-dimethoxy-4-(2-propenyl)- (2.01%), Dehydroabietic acid (1.03%), 2-Propenal,3-(4-hydroxy-3-methoxyphenyl)- (0.92%), 2(5H)-Furanone (0.89%), Ethanone, 1-(4-hydroxy-3,5-dimethoxyphenyl)- (0.75%), 2H-Pyran-2,6(3H)-dione (0.58%), 2-Furancarboxaldehyde, 5-methyl (0.37%).

According to research results show, 2-methoxy-Phenol is an important fine chemical intermediate, widely used in medicine, spices and dyes synthesis. 1-hydroxy-2-Propanone is the important component in volatile aroma of grilled lamb legs [10]. Dehydroabietic acid is widely used in coatings, adhesives, inks, paper, rubber, food additives and biological products and other fields. The use of dehydrogenated rosin structure characteristics, but also the development of high value-added new functional compounds [11]. 1-(4-hydroxy-3,5-dimethoxyphenyl)-Ethanone is a synonym for acetosyringone. In the plant explant culture, acetosyringone has a role in promoting the regeneration of roots and seedlings can promote plant conversion rate [12]. The current role of acetosyringone in monocotyledonous and dicotyledonous plants has been extensively studied. For example, acetosyringone addition can promote the transformation of celery and strawberry explants [13,14].

3.3 Analysis of TD-GC-MS

According to the results of TD-GC-MS analysis, 9 chemical constituents were identified in 34 peaks. Sorting the detected component by peak area ratio from high to low are: 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester (65.87%), Ethanol, 2-(2-butoxyethoxy)-, acetate (6.6%), 2,2,4-Trimethyl-1,3-pentanediol diisobutyrate (2.17%), Propanoic acid, 2-methyl-, 3-hydroxy-2,2,4-trimethylpentyl ester (1.67%), Benzene,1,2,3-trimethoxy-5-(2-propenyl)- (1.36%),1H-3a,7-Methanoazulene,2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3. alpha.,3a.beta.,7.beta.,8a.alpha.)]- (1.21%), 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester (1.13%), Dimethyl phthalate (0.87%), Linalool (0.6%).

From table 3 we can learn that 1,2-Benzenedicarboxylic acid, bis (2-methylpropyl) ester (diisobutyl phthalate) is far more than another component. Diisobutyl phthalate is the main plasticizer and softener used in the plastics industry. This component can be directly transferred to the food through the food packaging material and can be spread into the environment by the plastic product containing the substance, causing pollution to air, water, soil and food [15]. Toxicity studies are currently focused on reproductive developmental toxicity studies [16].

1H-3a,7-Methanoazulene,2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3.alpha.,3a.beta.,7.beta.,8a.alpha.)]- is alpha-cedrene. It is a very promising natural spices and chemical materials, can be used to produce a variety of drugs and fine chemicals [17]. Alpha-cedrene has a significant bacteriostatic effect on coriolus versicolor and gloeophyllum trabeum murr. Alpha-cedrene and the synergistic effect of antibacterial ingredients have a certain contribution to the antibacterial properties of the extract [18]. Dimethyl phthalate is a toxic organic compound that is widely present in the environment. The pollution from dimethyl phthalate can affect the diversity and metabolic function of microbial community [20]. It can be used as a plasticizer to improve the strength and plasticity of plastics [21], but also widely used in pesticides, insect repellent, paint and cosmetics [22,30].

Linalool is a volatile oil, it is perfume. It is the highest use of spices in perfume, household cleaning products, can also be used as edible flavor and important intermediates of industrial production [23,31]. In addition, linalool also has a health care function. Many domestic and international experimental studies have shown that linalool has analgesia [24], anti-anxiety [25], sedation [26], anti-inflammation [27] and anti-tumor [28], antisepsis [29] function.

3.4 Analysis of TG

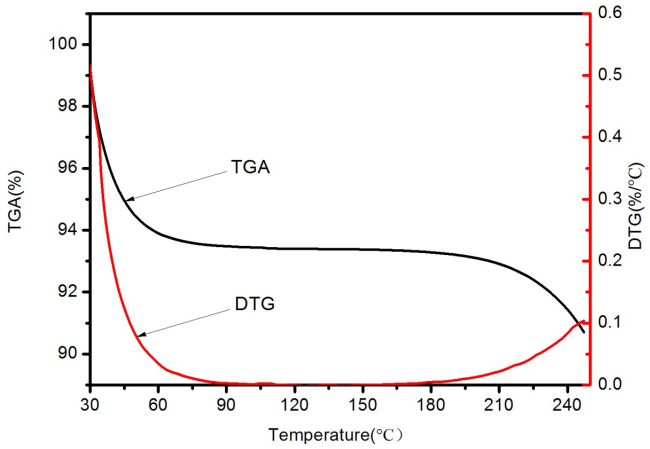

Figure 3: TGA and DTG thermal curves.

For analysis, we divide the DTG curve of Figure 3 into three stages. The first stage (T1) is 30℃ to 85℃ the second stage (T2) is 85℃ to 185℃,the third stage (T3) is 185℃ to 240℃. As can be seen the quality of camphor tree powder was decreased significantly. This phenomenon is due to the fact that the sample's bound water was evaporation at this stage. When the heating temperature is less than 100℃, the wood properties will not change significantly. Total dry mass of wood is only slightly reduced, is caused by microbial decomposition of hemicelluloses. In the second stage the curve tends to be gentle it is continuous endothermic process of wood. The quality of this process is reduced very few, indicating that the thermal stability of camphor is better. In the third stage, thermal degradation rate was raised. Because for most of the porous material, with the temperature rise, the thermal movement of its solid molecules will increase. The thermal conductivity of the pore air and the radiant energy between the holes wall are also increased, resulting the thermal conductivity of the material increase, cellulose and hemicellulose degradation, carbohydrate pyrolysis is easier, sample pyrolysis.

Table 1: Analytical results of FT-IR spectra.

Absorption peak(cm^{-1})	Absorption peak attribution	Chemical composition
3335	O-H Stretching vibration	Cellulose, Hemicellulose
2911	C-H Stretching vibration,	Lignin
1742	C=O Stretching vibration	Lignin, Hemicellulose
1597	Benzene ring stretching vibration	Lignin, Aromatic
1508	Benzene ring stretching vibration	Lignin, Aromatic
1464	Benzene ring stretching vibration	Lignin, Aromatic
1335	C-N Stretching vibration	Amine
1055	C-O Stretching vibration	Alcohol, Phenol, Ester

Table 2: Py-GC-MS analysis.

No.	Retention Time (min)	Peak Area (%)	Component
1	5.33	2.62	2-Propanone, 1-hydroxy
2	7.41	3.05	Propanoic acid, 2-oxo-, methyl ester
3	12.51	0.89	2(5H)-Furanone
4	13.06	2.34	1,2-Cyclopentanedione
5	14.47	0.37	2-Furancarboxaldehyde, 5-methyl
6	15.97	0.58	2H-Pyran-2,6(3H)-dione
7	19.77	3.93	Phenol, 2-methoxy-
8	29.05	2.29	Phenol, 4-ethyl-2-methoxy-
9	41.24	2.01	Phenol, 2,6-dimethoxy-4-(2-propenyl)-
10	41.70	0.75	Ethanone, 1-(4-hydroxy-3,5-dimethoxyphenyl)-
11	41.83	0.92	2-Propenal, 3-(4-hydroxy-3-methoxyphenyl)-
12	53.56	1.03	Dehydroabietic acid

Table 3: TD-GC-MS analysis.

No.	Retention Time (min)	Peak Area (%)	Component
1	7.675	0.6	Linalool
2	12.25	6.6	Ethanol, 2-(2-butoxyethoxy)-, acetate
3	12.426	1.67	Propanoic acid, 2-methyl-, 3-hydroxy-2,2,4-trimethylpentyl ester
4	13.145	1.21	1H-3a,7-Methanoazulene,2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3.alpha.,3a.beta.,7.beta.,8a.alpha.)]-
5	13.724	0.87	Dimethyl phthalate
6	15.211	1.36	Benzene, 1,2,3-trimethoxy-5-(2-propenyl)-
7	15.854	2.17	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate
8	19.597	65.87	1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester
9	20.114	1.13	1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester

4. CONCLUSIONS

The untreated camphor tree powder was used as experimental material detection by fourier transform infrared spectroscopy (FT-IR), thermogravimetry (TG), pyrolisis-gas chromatography-mass spectrometry (Py-GC-MS), thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS). Determination of chemical constituents of volatile oil from camphor tree powder and study its function and application value.

According to the results of FT-IR analysi, In volatiles of camphor tree powder there are cellulose, hemicellulose and lignin besides there are include alcohol, phenol, ester, aromatic, amine.

According to the results of Py-GC-MS analysis, 50 peaks were detected and 12 chemical constituents were identified and the peak area accounted for 20.78% of the total peak area. The detected component include: guaiacol (3.93%), methyl pyruvate (3.05%), hydroxyacetone (2.62%), cyclooctane-1,2-dione (2.34%), 4-Ethyl-2-methoxyphenol (2.29%), 4-allyl-2,6-dimethoxyphenol (2.01%), dehydroabietic acid (1.03%), 4-hydroxy-3-methoxycinnamaldehyde (0.92%), 2 (5H)-Furanone (0.89%), acetosyringone (0.75%), 2H-Pyran-2,6(3H)-dione (0.58%), 5-Methyl furfural (0.37%).

According to the results of TD-GC-MS analysis, 9 chemical constituents were identified in 34 peaks. Sorting the detected component by peak area ratio from high to low are: diisobutyl phthalate (65.87%), 2-(2-Butoxyethoxy)ethyl acetate (6.6%), 2,2,4-Trimethyl-1,3-pentanediol diisobutyrate (2.17%), Propanoic acid, 2-methyl-,3-hydroxy-2,2,4-trimethylpentyl ester (1.67%), Benzene,1,2,3-trimethoxy-5-(2-propenyl)- (1.36%), alpha-cedrene (1.21%), diisobutyl phthalate (1.13%), Dimethyl phthalate (0.87%), Linalool (0.6%).

Experiments of TG show that The thermal stability of camphor is good When the temperature is lower than 190 degrees, volatile or decomposed component only have water and component.

The volatile substances of camphor tree are very little. Areas of application include medical, paint, chemical and so on.

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